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Accelerated Adhesive Curing for Induction-Based Repair of Composites

by Steven H. McKnight, Bruce K. Fink, Sean Wells, Shridhar Yarlagadda, and John W. Gillespie Jr.

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Abstract

A methodology for accelerated curing of commercially available room-temperature curing paste adhesives is outlined. Cure kinetics of the adhesive were studied by thermochemical analysis, and degree of cure was related to processing parameters and cure cycles. Increasing the cure temperature to 100°C reduced the cure time from 16 hr to approximately 15 min for 98% cure. Induction-heating techniques were used to demonstrate rapid heating of adhesives at the bondline for lap shear specimens.

Acknowledgments

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1. Introduction

The motivation for this work arises from the expanding use and complexity of design of composites in military vehicles and, with that, the increasing need for field expedient and depot-level repair procedures for these components.

A critical issue in adhesive-based repair of composites is the application of sufficient heat and pressure at the bondline. It is highly desirable that thermal generation be localized at the bondline and be evenly distributed (taking into account thermal conductive losses). One method of rapidly applying localized heating at the bondline is induction heating [1].

Electromagnetic induction heating techniques are well known and widely used for metals and alloys. Recently, significant research has been undertaken to adapt induction heating to composites for benefits such as cost and reduced processing times. One of the heating techniques uses hysteresis losses in ferromagnetic particles subjected to high-frequency magnetic fields as the heat-generation mechanism. Another generates heat through joule losses caused by the formation of eddy currents through Faraday's Law. Both of these heating techniques can be applied to the repair of composites through the use of a susceptor material placed at the bondline or through the susceptorless heating of carbon-fiber-based systems [2, 3]. Susceptor layers are used to promote localized uniform heating to produce desired process temperatures in the bondline. When susceptors are used, the remotely located induction coil transfers electromagnetic energy to the susceptor, which in turn, generates thermal energy in the plane of the bondline. These techniques allow rapid heating of the susceptor material and, through thermal conduction, rapid heating of the adjacent adhesive. These methods have traditionally been plagued by nonuniformity of heating in the plane of the bondline. Several techniques have recently been developed [3, 4] that enable uniform heating of the susceptor in the plane of the bondline.

Appropriate process windows are needed for each adhesive system to be used. In this study, eddy-current-based susceptors are formed from electrically conductive meshes and an epoxy-based adhesive. Room-temperature curing adhesives that are often used in the repair of composites require from days to weeks to achieve full cure. This work establishes a methodology for relating cure cycles to degree of cure predictions for accelerated curing of adhesives for repair. Furthermore, the induction heating is used to accelerate the cure of a room-temperature curing epoxy adhesive placed at composite-to-composite bondline.

2. Thermochemical Analysis

In order to maximize the benefits of accelerated cure of adhesives using induction heating, a process window must be established for the adhesives of interest. The process window would then be used to optimize the bonding process in terms of time and temperature. Issues that dictate the process window include cure kinetics, evolution of exotherms, flow and wetting, and thermally induced residual stresses. Adhesive cure is the most dominant of these issues and must be addressed to determine cure time as a function of temperature, as well as ultimate degree of cure. In this study, we have chosen a typical room-temperature curing epoxy for evaluation of accelerated cure properties.

Differential scanning calorimetry (DSC) has been widely used to characterize the cure kinetics of thermosetting polymers including polyesters [5], epoxies [6], vinyl esters, and bismaleimides. Since the heat evolution dQ/dt measured by the DSC results from the chemical cross-linking reaction, it is possible to relate the heat evolution (dQ/dt) to the rate of reaction ($d\alpha/dt$) and the conversion (α). This can be accomplished by using the following relationships:

$$\frac{d\alpha}{dt} = \frac{1}{\Delta H_{tot}} \left(\frac{dQ}{dt} \right)_t , \qquad (1)$$

$$\frac{d\alpha}{dt} = \frac{1}{\Delta H_{tot}} \int_{t_0}^{t} \left(\frac{dQ}{dt} \right)_t dt , \qquad (2)$$

where ΔH_{tot} is the total heat of reaction, generally determined by averaging the reaction exotherms measured from several dynamic-temperature DSC runs. Various chemical kinetic models can then be fit using data that are obtained from isothermal DSC experiments.

The mechanistic models of thermoset cure that usually provide a more accurate representation of cross-linking reactions are not generally applicable to complex systems such as formulated adhesives. Since the goal of this work is to provide a process window for accelerated cure, the specific cure mechanisms need not be critically assessed. Alternatively, there are several empirical models that have been successfully used to predict to cure of thermosetting polymers. One popular model was proposed by Kamal and Sourour [7]. Their model (equation [3]) has found widespread acceptance for a number of cross-linking reactions (including epoxies) and will be used to fit the adhesive studied here.

$$\frac{d\alpha}{dt} = (k_1 + k_2\alpha^m)(\alpha_u - \alpha)^n \tag{3}$$

In this expression, α is the degree of conversion, α_u is the temperature-dependent maximum conversion, k_1 and k_2 are Arrhenius-type rate constants, and m and n are constants usually assumed to sum to 2 but often allowed to vary freely. The α_u term arises from the fact that the entire heat of reaction is not released during isothermal cure due to the decreased mobility of the polymer chains as cross-linking occurs. By performing a series of isothermal cures, values for the model parameters can be determined and used to predict the cure kinetics of the adhesive.

The material studied here was a two-part epoxy room-temperature-curing paste adhesive from Ciba. It was selected because of our prior experience with the system for composite and metal bonding. Additionally, the manufacturers suggest a 16-hr cure time at room temperature, making it an ideal candidate for accelerated cure studies.

Several (10) dynamic DSC runs were performed to evaluate ΔH_{tot} and the glass transition temperature (T_g) of the cured material. Resin and hardener were mixed one to one by weight and immediately inserted into the DSC (TA Instruments 2908), where they were heated at 10°C/min to 200°C. The resulting cure exotherm was integrated to evaluate the heat of reaction. A second heat of each sample was performed in order to measure the T_g of the cured material. The average and standard deviation of ΔH_{tot} was 190.5 ± 10.2 J/g and T_g was 102 ± 14°C. This value of ΔH_{tot} is used in equations (1) and (2) to relate the isothermal heat data to α and $d\alpha/dt$.

Next, isothermal scans were performed at temperatures ranging from 40°C to 150°C. Samples were mixed and placed in the preheated DSC cell. Data were collected until the heat flow returned to the baseline value. The isothermal heat flow was related to α and $d\alpha/dt$ using equations (1) and (2). A typical DSC isotherm and the resulting conversion vs. time are shown in Figure 1. Figure 2 shows the general trend of increased conversion and rate of reaction with increasing cure temperature.

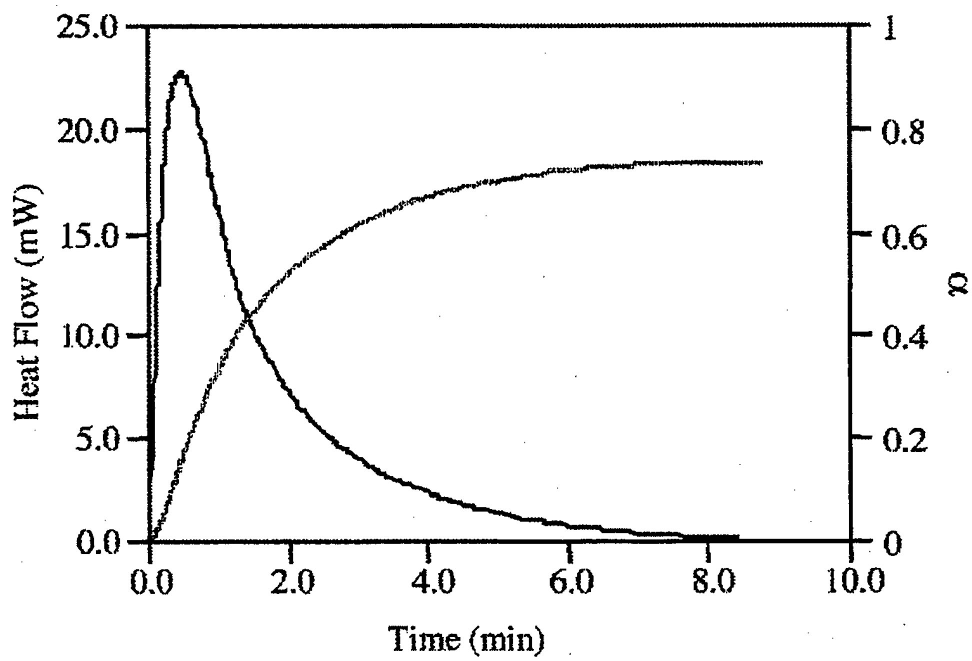

Figure 1. DSC Heat Flow for Isothermal Cure at 110°C and the Associated Degree of Conversion With Time.

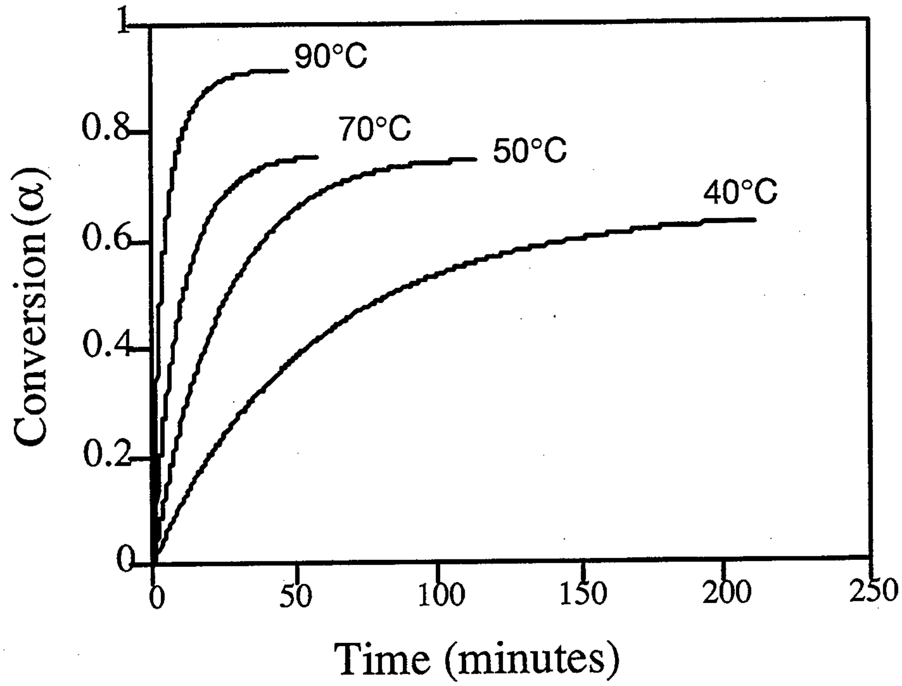

Figure 2. Degree of Conversion Versus Time for Increasing Isothermal Cure Temperature.

Equation (3) was then used to fit the $d\alpha/dt$ versus α curves for each isotherm. A value of α_u was determined from the asymptotic conversion from each test, and m and n were permitted to vary freely. Figure 3 shows typical data and the associated fit. Analysis of each experiment produces values for all of the kinetic parameters at that specific temperature. The temperature dependence of α_u was found to be linear and is shown in Table 1. The Arrhenius parameters for k_2 were evaluated as shown in Figure 4 (analysis of the data indicated that $k_1 \approx 0$ regardless of temperature and was subsequently neglected). A summary of all of the parameters is listed in Table 1.

The use of the model will enable prediction of the entire curing process over a wide range of processing temperatures. Initially, however, the prediction of cure time at a specific temperature is of greatest interest to applying induction techniques to accelerate adhesive cure. Here, cure time is defined as the amount of time necessary to reach 98% of α_u for each temperature.

Table 1. Kinetic Model Parameters

Parameter	Value
m	0.28 ± 0.03
n	1.67 ± 0.32
$k_2(T)$	$9.8 \times 10^6 \exp(-6306/T\ [K])$ (1/min)
$\alpha_u(T)$	$0.62 + 1.3 \times 10^{-3}\ T\ (°C)$ $(40 < T < 150°C)$

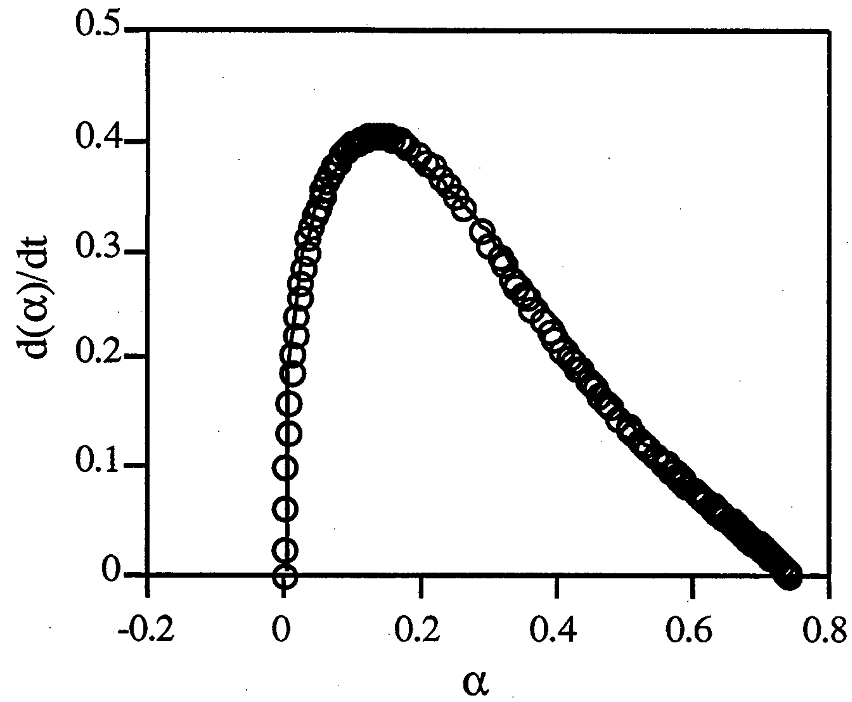

Figure 3. Plot of dα /dt Versus α for a Typical Isotherm With Associated Fit.

Figure 5 shows model predictions for cure time compared to the experimentally observed cure times. While the agreement is not perfect, it does permit an estimate of minimum cure time at each temperature. These values will be used to determine process windows for the induction assisted accelerated cure of this adhesive.

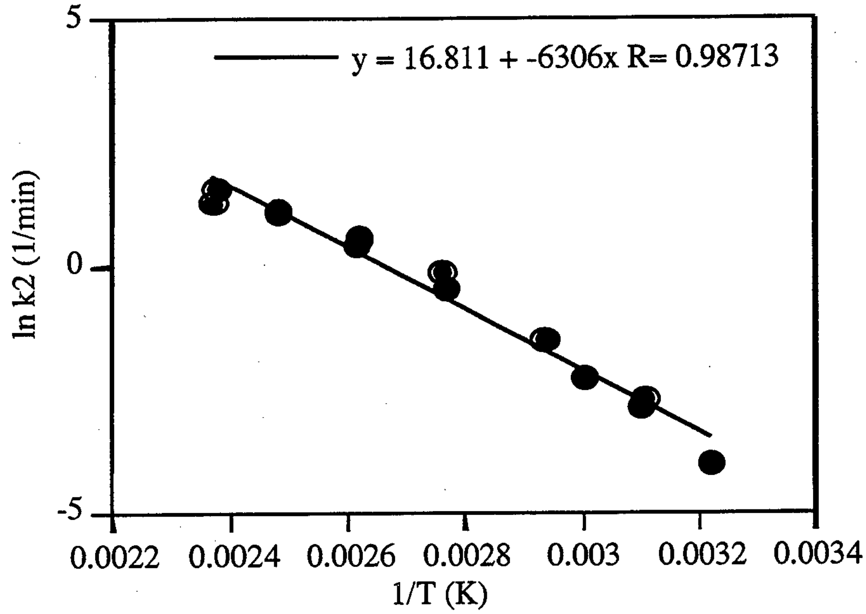

Figure 4. Arrhenius Relationship for the Parameter k_2 Used in the Kinetic Model.

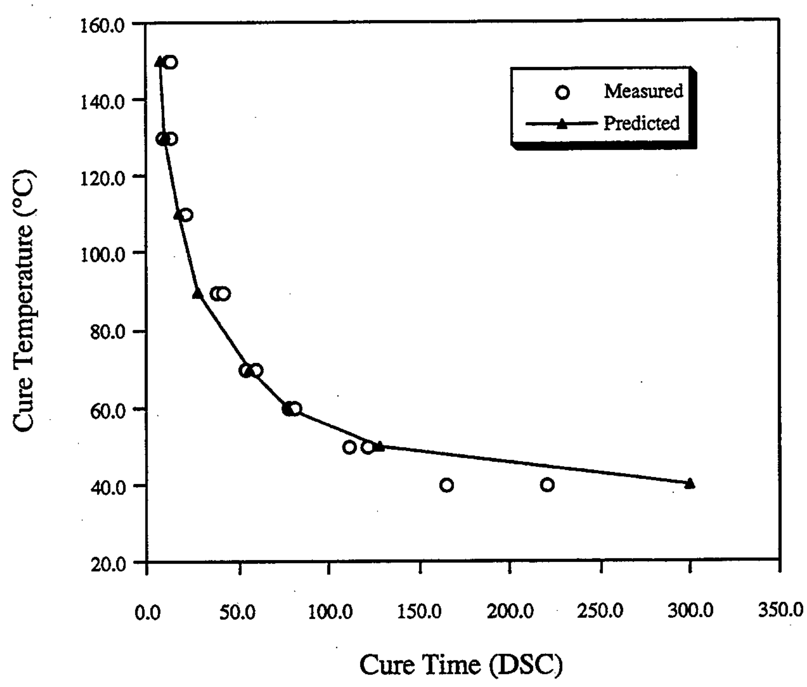

Figure 5. Model Predictions for Cure Time Compared to Experimentally Observed Cure Times.

3. Induction Heating

Appropriate cure times for this adhesive can now be selected for any process temperature. This approach was used to select cure cycles for induction heating of the composite adhesive joints. Cure cycles chosen ranged from heating to 90–190°C under vacuum consolidation. Single lap shear specimens were fabricated by induction heating using a stainless steel mesh as the susceptor. An "earmuff" type induction coil was used and it carried currents between 25–40 Amps at a frequency of 284 kHz. Typical temperature profiles during induction heating of lap shear specimens are shown in Figures 6 and 7.

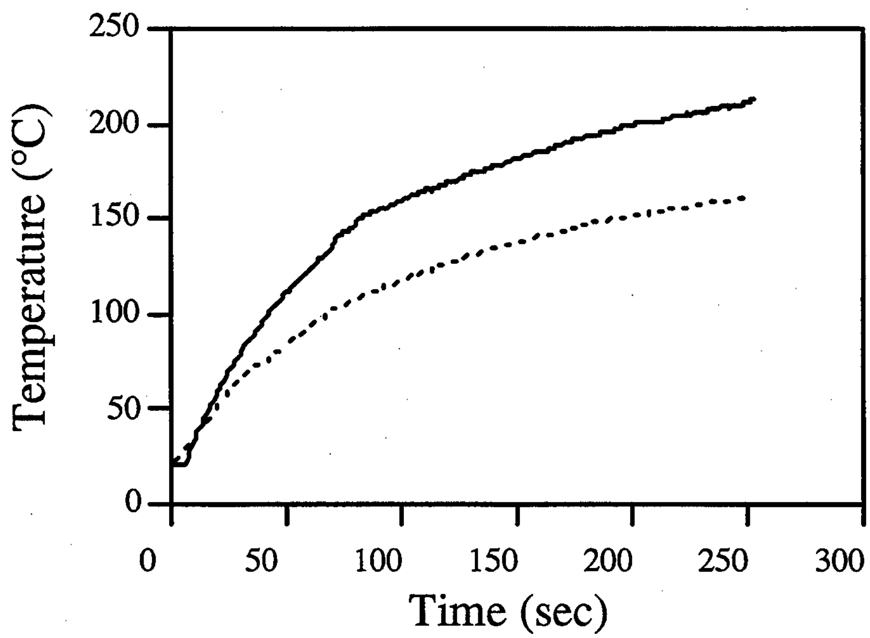

Figure 6. Typical Temperature Profiles for Induction Heated Adhesive Joints. T_{max} = 150°C (Dotted Line) and 205°C (Solid Line).

For baseline comparisons, lap shear specimens were fabricated under oven cure conditions with vacuum consolidation. Lap shear tests showed comparable bond strengths between induction-fabricated specimens and oven-cured specimens.

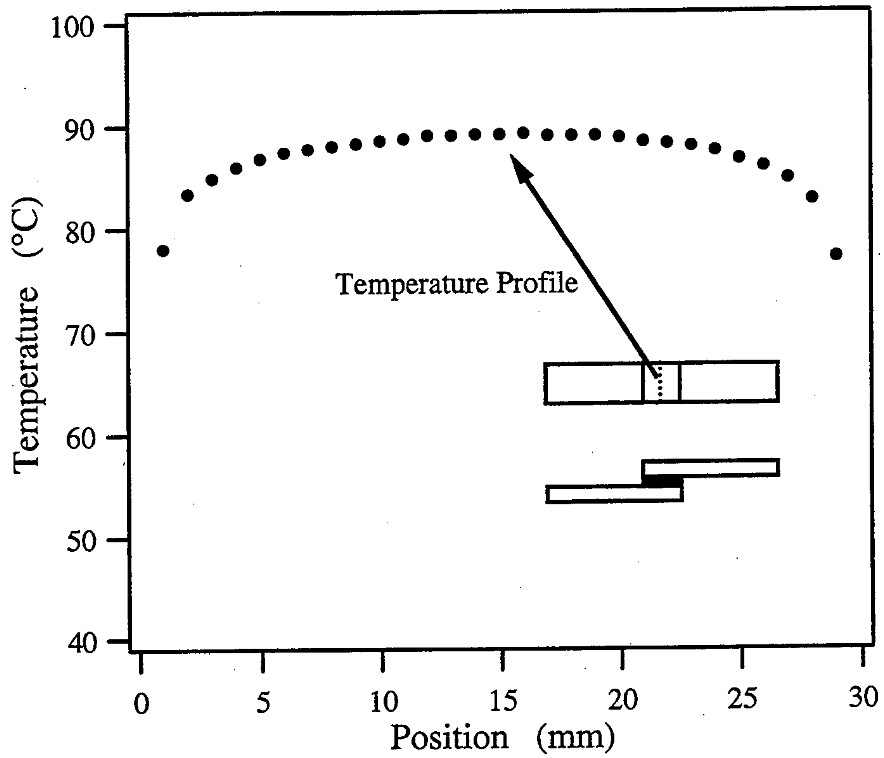

Figure 7. Typical Measured Temperature Profile at Bondline at Steady State by Infrared Thermometry.

4. Conclusions

This report has described a methodology that can be used to accelerate the cure of room-temperature curing adhesives for rapid repair. Cross-linking reaction kinetics were developed and employed to determine cure cycles for a commercially available epoxy paste adhesive. This paste adhesive was combined with a metal screen to form a susceptor layer for bonding composite adherends. Induction techniques were used to rapidly heat the interface and cure the adhesive. Adhesive taken from the bondline demonstrated full cure at times determined from the kinetic models.

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A methodology for accelerated curing of commercially available room-temperature curing paste adhesives is outlined. Cure kinetics of the adhesive were studied by thermochemical analysis, and degree of cure was related to processing parameters and cure cycles. Increasing the cure temperature to 100°C reduced the cure time from 16 hr to approximately 15 min for 98% cure. Induction-heating techniques were used to demonstrate rapid heating of adhesives at the bondline for lap shear specimens.

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